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Massachusetts Inst of Tech Cambridge Lab for Insulat—ETC

Dielectric Spectroscopy of High-Temperature Materials.

Westphal, W. B.

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Errata Sheet

- p. 11, Program 1, lines 4 and 5 from top have to be retyped so that cosine is moved 3 spaces to the right.
- p. 21, IT. MISCELLAMEGUS INORGANICS AND MIXTURES, the tabulations should read:

Concrete pavement

California Highway Department

Sample	Density (g/cm ³)	H ₂ O (%)	(MHz)	0.1	1	10	100
Sl	2.284	0	x	9.05	7.97	7.01	6.57
			tan δ	.0946	.0913	.0730	.0536
S1		25.8 9	K	176.5	69.2	23.5	13.2
			tan δ	.822	1.088	.734	.485

Asphalt pavement

California Highway Department

Sample	Density (g/cm ³)	H ₂ O (%)	(Hz)	10 ⁵	10 ⁶	107	108
S	2.06	0	K.	4.51	4.34	4.2J	4.14
			tan δ	.0280	.0221	.0181	.0198
S		2.55	κ	42.0	17.7	9.03	6.54
			tan δ	.875	.638	.444	.233
L	2.46	0	κ	4.79	4.73	4.70	4.61
			tan δ	.0187	.0158	.0123	.0121
Ĺ		~0.36	κ	14.48	9.28	6.65	6.01
			tan δ	.368	.280	.190	.104

Dielectric Spectroscopy of High-Temperature Materials

W. B. Westphal and J. Iglesias

Laboratory for Insulation Research Massachusetts Institute of Technology Cambridge, Massachusetts

The distribution of this report is limited because of data and information related to a technology restricted by U.S. Export

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FOREWORD

This report was prepared by the Massachusetts Institute of Technology, Laboratory for Insulation Research, Cambridge, Massachusetts, under USAF Contract F33615-70C-1220. This Contract was initiated under Preject No. 7371, "Exploratory Development in Electrical, Electronic, and Magnetic Materials," Task No. 737101, "Dielectric Materials." The work was administered under direction of the AF Materials Laboratory, with Mr. W. G. D. Frederick acting as project engineer.

This Final Report covers work conducted from February 1, 1970 to January 31, 1971, and was submitted on February 26, 1971 by the authors for publication.

This technical report has been reviewed and is approved.

Charles E. EHRENFRIED

Major, USAF

Chief, Electromagnetic Materials Br.

Materials Physics Division

Air Force Materials Laboratory

ABSTRACT

and loss measurements to liquid-nitrogen temperature at 10 MHz, to 2000°C at 100 MHz, and to 1600°C at 90 GHz are discussed. High-temperature measurements on spinel and sapphire are included in the dielectric data accumulated during this contract. Programs in Fortran IV are given for the general standing-wave method calculations and for covered high-loss samples one-quarter wavelength from the end of hollow waveguide.

TABLE OF CONTENTS

	Page
Introduction	1
Measurement and Instrumentation Techniques	1
Low-Frequency Bridges	1
extension for a Two-Terminal Bridge	3
Resonant Cavity for 100 MHz	4
Resonant Cavity Method for Soft Materials	6
90-GHz Equipment	7
Programming	8
References	10
Program I	11
Program II	13
Index to Dielectric Data	16

ILLUSTRATIONS ...

Figure		Page
1.	Schematic of precision low-frequency bridge.	2
2.	Photographs of bridge switch.	3
3.	Photographs of bridge extension: (a) for above	3
	ambient; (b) for below ambient.	
4.	Reentrant cavity: (a) cross section; (b) photograph;	5
	(c) symmetry plane; (d) equivalent circuit of	
	reentran cavity.	
5.	Equipment for measurement of Q of reentrant cavity.	6
6.	High-temperature pressure welding of resonant cavity;	6
	(a) foil formed on mandrel, two are made. Faces are	
	welded by hot-pressing; (b) welding of flanges by	
	hot-pressing with sample inside; (c) finished dielectr	ic-
	filled cavity; (d) cavity in graphite container as	
	used during measurements.	

INTRODUCTION

an the first section of this report extensions of measurement techniques developed since March 31, 1970, are described. These include a method for using the dielectric-filled cavity technique with soft materials, details of design for a 100 MHz reentrant cavity, and the use of a light pipe for 3-mm measurements. The next section discusses programming for dielectric calculations. The final section lists measurement data on materials and is supplementary to AFML-TR-70-138.

MEASUREMENT INSTRUMENTATION AND TECHNIQUES

Low-Frequency Bridge

Acquisition of a General Radio Type 1620-AP capacitance measuring assembly has allowed us to recalibrate the precision capacitor used in our two-terminal bridge for high-temperature measurements. The basic accuracy of 0.01% assures that capacitance measurement errors on solid samples will be negligible in comparison to thickness measurement errors.

Experience in measuring small (<2 pf) high-loss, three-terminal samples has indicated the need for more precise measurements than can be obtained with any known direct-reading bridge. For example, the accuracy in measuring a 1-pf, 1-megohm sample at 100 cycles is limited as follows:

The GR 1615 can be balanced only with M=10 or greater which limit capacitance resolution to 0.01 pf (1%). In addition, capacitance of the conductance (G) network causes an unspecified error.

On the GR 1616, the capacitance resolution is not a significant error, but the specified possible error due to the capacitance of the G network is 0.03 pf (3%).

On our present laboratory bridges¹⁾ and the Cole-Gross bridge²⁾ errors due to the capacitance of the G network can be made small only by a sacrifice in sensitivity by increased loading of the detector terminals.

The Harris bridge avoids the above problems by using a two-phase source but the practical limit of air capacitors in the G phase is about 1000 pf, corresponding to a sample resistance of 1.6 megohms minimum at 100 cycles (if full voltage is applied to the sample). 3)

A new bridge is under construction which should provide routine operation with capacitance errors due to the C network of 0.601 pf and provides an adjustment to reduce the error toward zero for any sample with extra bridge balances. The bridge shown schematically in Fig. 1 is designed for use in the frequency range 0.5 Hz (with 1 volt) to 1 kHz. Six digit resolution in C is provided by 2

Work described in this section was jointly sponsored by ONR (Contract NO0014-67A-0204-0003).

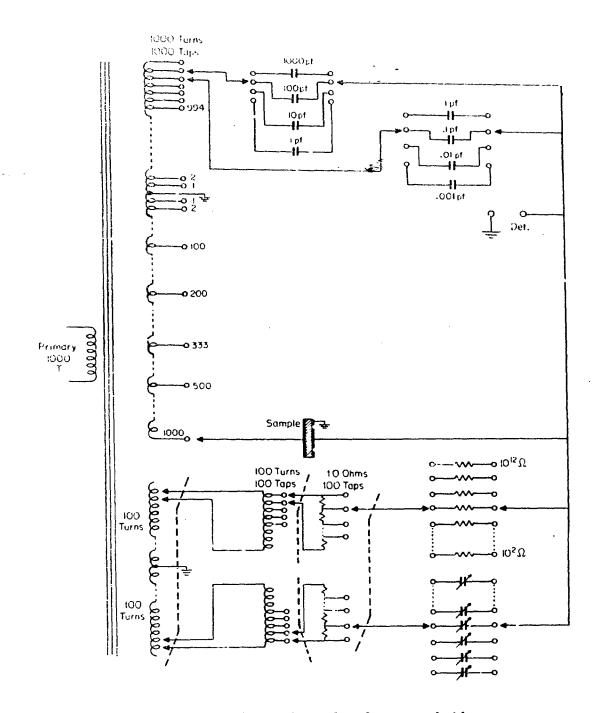


Fig. 1. Schematic of precision low-frequency bridge.

digit resolution in G is provided by using 100 turns of the transformer plus a 100-turn divider transformer plus a resistance potentiometer. These controls are duplicated with a symmetrical construction in the sample side of the bridge; here capacitors are adjusted to balance out the capacitance of the opposite conductivity network. Assuming that the transformer and C networks are ideal, successive balances made using phase-sensitive detectors and a series of samples with increasing loss should lead to a properly compensated G network. In Fig. 2, the



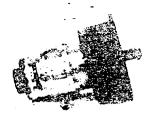


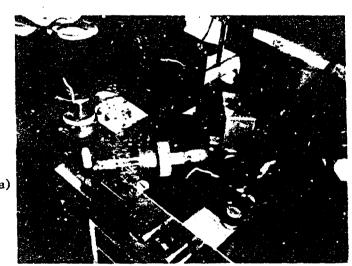
Fig. 2. Photographs of bridge switch.

construction of the 1000 point, 2-circuit capacitance switch is shown in present state. Contact fingers not shown will mount on the rotating cage and will contact plus in the phenolic drum. The holes for these pins are shown in a spiral pattern,

10 turns, 100 points per revolution.

Extension for a Too-Terminal Bridge

A basic problem exists in using our too-terminal wide-range bridge⁴⁾ for both hot and cold temperatures. For high temperatures the coaxial line connecting the sample to the bridge should go upward from the bridge to avoid heat transfer by convection to the bridge; for cold temperature the sample should be below the bridge. If separate sample holders are used for the two temperature regions, the sample must be transferred with risk of moisture coatamination and two separate calibrations of sample holder capacitance are required. To avoid this problem, we have comptructed an extension with a curved joint in a borizontal line. ... shown in Flg. 3, ordinary copper tube firtings, 2-1/2 inch size, were used for the outer condector.



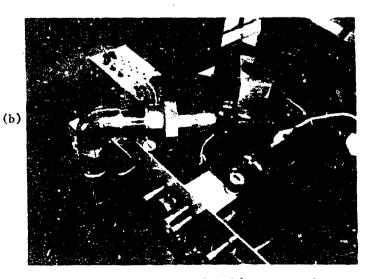


Fig. 3. Photographs of bridge extension:
(a) for above ambient; (b) for below ambient.

The inner conductor size is 7/8 inch. These large conductors, combined with close proximity of soften and sample, result in nominal line loss correction of 0.0002 in tan 6 at 10 Miz.

Resonant Cavity for 100 MHz

As indicated in a previous report, $^{5)}$ a high-temperature measuring system at $^{100~MHz}$ would be a desirable addition in the temperature—frequency plot of this laboratory's capabilities. A doubly-reentrant cavity (Fig. 4a) offers the possibility of reasons by accurate loss measurements to 2000 °C. Direct measurement of absolute value of κ ' might not be accurate (because of expansion effects in the cavity), but comparisons between materials should be satisfactory. The equivalent lumped circuit of the cavity is visualized by cutting the outer conductor on the center plane. The shorted coaxial line sections at each end are less than $\lambda/4$ long; at one frequency their equivalent is a lumped inductor (Fig. 4d). The center capacitor is divided into two sections by the center plane; each capacitor has the value 2C. Resonance obviously occurs when $2\omega^2 LC = 1$ in the equivalent series circuit when the outer conductor is reconnected. Calculations of theoretical losses for an empty cavity show the losses separated into 4 parts:

- 1. Resistance of outer conductor of coaxial line.
- 2. Resistance of inner conductor of coaxial line.
- 3. Resistance of shorting plane at end of line.
- 4. Resistance of electrodes.

In Table 1 are tabulated the results of calculations for 3 cavities, all copper at 25°C, inner conductor and electrode face of Pt at 25°C, and the same at 1600°C. The measured value of Q for the copper cavity includes the contact losses of the spring fingers joint, coupling losses to generator and detector, and dielectric loss in the heat-shielded material; the 25°C, 100-MHz value is 4350. Figure 5 shows the equipment used for determining Q by the frequency variation method.

Table 1. Losses in recentrant cavity at 100 MHz, $C_g = 33.9 \text{ pf}$.

Conductors	R1	R2	R3	R4	Q
Copper, 25°C Part Cu, part Pt, 25°C " " 1600°C Copper,* 25°C * At 150 MHz C _S = 14.6 pf.	0.22	0.92	2,61	0.10	6100
	0.22	2.27	2,61	0.51	4175
	0.22	6.00	2,61	1.34	2305
	0.43	1.79	3,20	0.26	6400

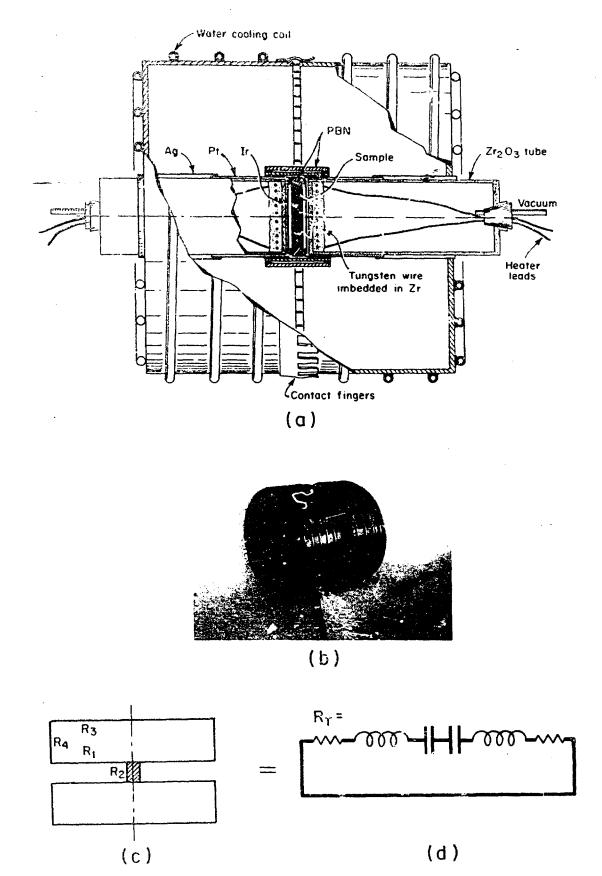
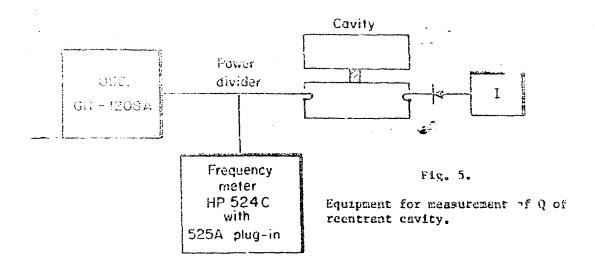


Fig. 4. Reentrant cavity; (a) cross section; (b) photograph; (c) symmetry plane; (d) equivalent circuit of reentrant cavity.



Resonant-Cavity Method for Soft Materials

Our usual procedure for measuring materials above 1000°C has been to form a dielectric-filled cavity of the sample by wrapping it with Pt or Pt-Rh foil and

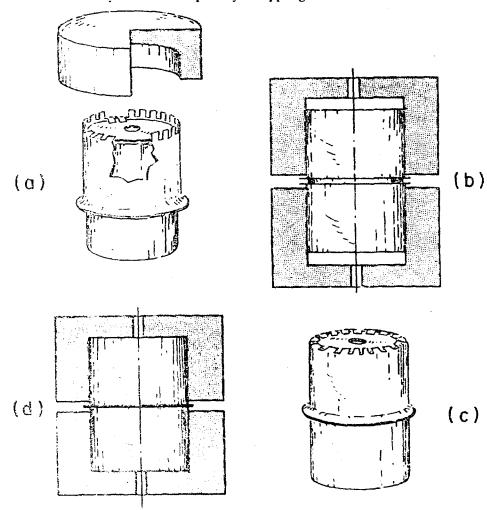


Fig. 6. High-temperature pressure welding of resonant cavity; (a) foil formed on sandrel, two are made. Faces are welded by hot-pressing; (b) welding of flanges by hot-pressing with sample inside; (c) finished dielectric-filled davity; (d) cavity in graphite container as used during measurements.

welding the foil by hot-pressing against the sample faces. An alternate method is to make two flanged cups as shown in Fig. 6 using graphite plunger and die. With the sample in the cups, the rims are hot-pressed together. As usual, graphite cups surround the sample during measurement.

90-Gliz Equipment

This bridge equipment, so originally built, was intended for use with relatively thin samples (<1 cm) and small diameter (5/8 inch), with an absorbing tube surrounding the sample. Measurements on fused silica and pyrolytic graphite have not yielded measurable loss values. Longer samples showed attenuation which varied depending on the absorber; this indicates the beams of energy are not narrow enough for long samples. Metallic tubes were substituted but have not been fully evaluated to find minimum satisfactory diameter and length combinations.

PROGRAMMING

Computation of K', K" in Standing-Wave Measurements

The computation procedure for covered samples less than 1.4 radians long and mounted over a quarter wavelength in coaxial line has been previously given. 6) The program has been developed for any length of sample in hollow waveguide with TE mode. The same program may be used for a sample without cover (by setting the constants K1 and K2 equal to zero) and with coaxial line [by setting $(\lambda/\lambda_c)^2$ equal to zero]. The input data consist of node width with sample-in corrected for loss in air-filled section of line (DS), the node position with sample-in (SN) and the node position with sample out (AN). The value of $2\pi X_0/\lambda$ in the program is written

$$2\pi[XE - AN + SN(I)]/LW$$

There are three common ways of taking the sample-out node position:

- (1) Sample and cover are both removed, so empty holder is used. Then XE = -(D2 + D3), where D2 and D3 are the thickness of cover and sample, respectively.
- (2) Sample is removed, cover is left in same position. Then XE is the calculated value of X, with cover and empty sample space:

$$XE = \frac{\lambda}{2\pi} \tan^{-1} \left[\frac{K1 - \tan(2\pi D3/\lambda)}{1 + K2 \tan(2\pi D3/\lambda)} \right].$$

(3) Sample holder (in which the top of the cover is located a distance \mathbf{d}_h from the top of the holder) is removed and replaced by a shorting plane. Then $XE = \mathbf{d}_h$.

When the sample over quarter wavelength holder is used as in the previous program,

$$XE = \frac{\lambda}{2\pi} \quad \tan^{-1} \left[\frac{\cot(2\pi D3/\lambda) + K1}{1 - K2 \cot(2\pi D3/\lambda)} \right].$$

In the programs and in the above empressions:

 λ = LW = wavelength in air-filled section of waveguide [hollow (TE modes) or coax (TE mode)],

FC =
$$\left(\frac{\lambda}{\lambda_C}\right)^2$$
,

$$W = 1 + FC$$

$$K1 = -\frac{1}{(\kappa_2^* W - FC)^{1/2}} \tan \left[\frac{2\pi D2}{\lambda} (\kappa_2^* W - FC)^{1/2} \right],$$

$$K2 = -(\kappa_2^* W - FC)^{1/2} \tan \left[\frac{2\pi D2}{\lambda} (\kappa_2^* W - FC)^{1/2} \right],$$
 $K3 = \frac{\lambda}{2\pi D3}.$

Losses in the cover are neglected but the program can be readily changed to include these losses if necessary. The wall losses in sample-filled section are included in the results as computed. They are readily subtracted at the end of the computation. 6)

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 Ice, and Aqueous Solutions and Their Interpretation. VI. New Measurements
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- Reference 5, pp. 6-9, 14,15.
- 7. Reference 2, p. 86.

PROGRAM I

THIN COVERED SAMPLE (<1.4 radians) OVER QUARTER WAVELENGTH IN HOLLOW GUIDE

```
FORTRAN IV G LEVEL 18
                                         MAIN
                                                             DATE = 71115
                                                                                    11/59/03
 0001
                    INTEGER#4 I.J.K.N.NX
                    REAL*R K1.K2.K3, Y.XE.AN.SN.LW.DX.DS.DA, X.A.B.A2, 82, 214RE, 714IM.
 2002
                   ?83LD+Z14IAD+Z14R9D+ERROR1+EROLD+A0LD+AIM+RE+TAM,Z11RE+Z11IM+WE+
                3COS [NE+FC+W,ZM,Y1,TAHA,TANB,PII,PII2,ZR3,ONE,STEP(4)/1.D-2,1.D-3,
                   41.0-4,1.0-5/.ND8.F(2)/1.00,-1.00/
 0003
                    COMPLEX*16 Z1.Z2.Z3.Z4.Z5.Z6.Z7.Z8.Z9.Z10.Z11.Z12.Z13.Z149.Z141.
                   2214, Z12NEW, Z12RE, Z12IM, Z12SQ, G, H, Z15, Z2A, ZONE, ZOONE
 2004
                    REAL*B SILLY(2), FAKE(2), FUN(2)
 2005
                    FQUIVALENCE (SILLY(1), Z11), (FAKE(1), Z15), (FUN(1), Z12SQ)
 2206
                    DIMENSION DS(19).SN(19).DATE(18)
 0007
                    NAMELIST/IN/DS+SN+NX/CONST/DA+AN+XE+K1+K2+K3+FC+H+LH/DUT/K1+K2+
                   2K3+LW/DUT2/21+72+24+29+A+B
 0008
                200 FORMAT(1X-18A4)
 0009
                 77 READ(5,200,END=88) DATE
 0019
                201 FORMAT(1H1,20X,1844)
 2211
                    WRITE(6,201) DATE
 0012
                    READ(5.IN)
                    READIS, CCNSTI
 9913
 2014
                    WEITE(6. IN)
                    WRITE (6.CONST)
 2015.
 2016
                100 FORMAT(1H0,5%,2HNS,10%,2HDS,11%,2HK1,11%,2HK2,12%,3HTAN,20%,3HZ11,
                   231X,3HZ14//)
 0017
                    WRITE(6.100)
                    ZONE=(1.D0.0.D0)
 0018
                    ZOONE = (0.00.1.00)
 0019
                    PII=31416.0-4
 2020
                    PI12=62832.D-4
 0021
 0022
                    ZRO=0. DO
 2023
                    ONE=1.00
 0024
                    00 10 1=1.NX
                    IF (SN(I). EQ. AN) GO TO 10
 9925
                    Y=P112*(XE-AN+SN(1)1/LW
 0026
 2027
                    DX=DS(I)-DA
                    IFIDX.LE.ZROI GO TO IP
 0028
 2029
                    COSINE=DCOS(PII+OX/LW)**2
                    X=DSIN(PII*DX/LW)/DSQRT(2.DD-CDSINE)
 2039
 0031
                    Y1=CTAN(Y)
 0032
                    Z1 = Z30NE*Y1
 0233
                    ZZA=ZONE*X
 0034
                    Z2=Z2A-Z1
 0035
                    23=20NE-22A*21
 0036
                    24=22/23
 0037
                    7.5=200NE*K1
 2038
                    26=24+25
 0039
                    27=2 )DNE + K2
                    Z8=ZONF+Z4*Z7
 7740
                     29=28/26
 0041
                     210=200NE/K3
 2042
 0043
                     711=710+79
                     Z11RE = SILLY(1)
 0044
 0945
                     Z111M=SILLY(2)
                     Z12SQ=CDSQRT(Z11+(1,D0/3,D0)*Z11**Z)
 0046
 2047
                     A=FUN(1)
                     B=FUN(2)
 2048
 0049
                 160 TAHA=CTANH(A)
                     TANS=DTAN(8)
 9050
                     AZ=TAHA#{ONE+TANB##2}/(ONE+TAHA##2#TANB##2)
 0051
 0052
                     B2=TANB=(CNE-TAHA**2)/(ONE+TAHA**2*TANB**2)
                     Z14RE=A=A2-B=B2
 0053
 0054
                     2141M=A*87+8*A2
                     ERPORT = 050RT((Z14RE-Z11RE) ** 2+(Z14 T4-Z11T4) ** 2)
  2055
  0056
                     DJ 430 K=1,4
                     TAHA= DTANH( 1)
  0057
  0058
                     50 500 J=112
                     WE=OYE+STEP(K)*E(J)
  0059
  0060
                 401 BOLD=8
  0061
                     Z1410D=Z141M
                     714ROD=214RE
  0162
  2063
                     EROLD=ERROR1
```

PROCRAM I (cont.)

```
3064
                   BEHFUE
~n65
                   TANB=DTANES;
1066
                   A2=YAHA#(PNE+TANB##21/13NE+TAHA##2#TANB##2)
3767
                   BZ=TANB* (ONE-TAHA**2)/(ONE+TAHA**2*TANB4*2)
9969
                   214EE=A#A2-B#82
3069
                   7141M=A=B2+B=42
9079
                   FRROK1=DSORT!(Z14RE-Z11RE)**2+(Z141M-Z111M)**2)
10.71
                   IFIERRORI.LE. EROLDI GO TO 401
0072
                   2141M=214100
0073
                   214RE=214R90
0074
                   B=BOLD
9675
                   ERROR1=EROLD
               600 CONTINUE
9476
2077
                   TANR=DTAKES)
re78
                   00 700 J=1,2
9079
                   WE=ONE+STEP(K)+F(J)
0389
               402 40LD=A
2800
                   Z14ROU=Z14RE
1082
                   Z1418D=Z141M
3093
                   FROLD=FRRORT
2084
                   G=A*NF
1085
                   TAHA=DTANH(A)
1986
                   AZ=TAHA*(CNE+TANB**?)/(ONE+TAHA**?*TANB**2)
1087
                   82=TANB*(ONE-TAHA*+2)/(ONE+TAHA**2*TANB**2)
วดอล
                   Z14RE=A* A2-B*B2
0089
                   21414=A+82+8+42
3091
                   FRRUR1=DSORT({Z14RE-Z11RE}*2+(Z141M-Z1114)**2)
2091
                   IF (ERRORI.LE. FROLD) GO TO 402
ባባዓን
                   714RE=214900
                   2141M=214100
1/03
2094
                   4=AOLD
2295
                   EKROR1=FPOLO
2195
               JUNITHCO CONTINUE
1197
                   IF(ERROR1.LE.1.D-6) GO TO 450
RP 10
               400 CONTINUE
               450 Z12RE=ZONE*A
(199
                    Z12IM=ZOCNE#B
4103
2121
                   712NEW=(212RE+2121M) **2
                                                   NOT REPRODUCIBLE
0102
                   213=-212NF#*K3**2
0103
                   G=ZONE*FC
0104
                   H=ZONE*W
9105
                   215=(G+Z13)/H
                   714R=20NE*Z14R5
0106
                    Z14I=Z00NF+Z14IM
2127
                    Z14=Z14R+Z141
0109
0100
                    RE=FAKE(1)
                    AIM=-FAKE(2)
2110
                    TAM=AIM/RE
^111
               317 FORMAT (2X.F8.4.5X.F7.4,5X.F9.3.5X.F9.3.5X.F8.4.5X.E13.6.3X.F13.6.
 1112
                   25X.E13.6.3X.E13.6)
                    #RITE(6,300) SN(1).DS(1).RE,AIM.TAM,Z11.Z14
1113
                    WRITE(6.CUT2)
2114
0115
                 10 CONTINUE
0116
                    90 TO 77
2117
                 88 CALL EXIT
                    END
 1118
                                     TYPICAL PRINT-OUT
          NS
                       DS
                                     ĸ1
                                                   K2
                                                                  TAN
        0.6250
                     0.0700
                                    30.705
                                                    4.965
                                                                 0.1617
        0.6310
                     0.0710
                                    29.533
                                                    4-894
                                                                 0.1657
                              211
                                                                  214
                -6.2717080-31
                                  0.4506940-02
                                                    -0.2717070-01
                                                                      0.4506930-02
                                  0.4439130-02
                                                    -0.2610690-01
                -0.251066D-01
                                                                      0.4439150-02
       K1=-0.1131899999999999
                                   *K2= -1.198499999999999
                                                                      46.9949999999999
                                                                .K3=
       EFND
```

12

+LW= 4,499999999999999

PROCRAM II

COVERED SAMPLE OF ANY LENGTH (A MALE WAVELENGTHS) ACAINST SHORT IN HOLLOW COIDS

FORTRAN	TV C (EVEL 18	MAIN	DATE = A111	2 17/37/44
6601	Integra 44	I.J.K.N.NX		
0302	REAL*8 K1	.K2.K3.Y.XE.AN.SN.EW.DX	.OS.DA.X.A.B.A2.B	2,214RE,214IM,
	2831.0+2141	OD.Z14ROD.ERROR1.EROLD.	AOLD-AIM-RE.TAM.Z	11RE.Z111M.WE.
	3COS TNE+FC	.W.7M.YI.TAHA.TANB.PII.	PIII2.ZRO.DNE.STEP	(221/0-100.5-D 2.
		-2.5.0-3.2.0-3.1.0-3.5.		
	55.0-6.2.0	-6.1.D-6.1.D-7.1.D-8.1.	D-9.1.D-10.1.D-11	.1.D-12/.ND8.
	6F(2)/1.00	1.DO/.SOLD		
0603		6 21.12.23.24.25.26.27.	28.29.210.211.212	.Z13.Z14R.Z14I.
		W.ZIZRE.Z12IM.Z1250.G.H		
0694		LLY (21. FAKE (2)		
2005		CE (SILLYEI).ZAI).(FAK	E(1).215)	
6036		DS (56) . SN (56) . N (56 1. DA		
0607		IN/DS.SN.N.NX/CONST/DA.		.W.LW/OUT/A.
		141M.K.J.ERROR1.KOUNT.W		
ocon	200 FORMAT (1X		_	
0009		C.FND=88) DATE		
0010	201 FORMATEIH			
0011	WRITE(6.2			
0012	READ(5. IN			
	READUS.CO			
0013	WRITE 16.1			
0014	WRITE(6.C			
0015		0.5X.2HNS.6X.2HDS.6X.1H	M CY 2011.04 2MY2	QY - 241YAN -
3016			M+ 0x + 5 HK I + 7X + 21 K2	
		.28X.3H714//i		
0017	WRITE(6.3			
0018	ZONE=(1.0			•
0019	ZOONE=(O.			
0^20	P11=31416	=		
0021	PI12=6283	2.D-4		
0022	₹80 =0. 00			
0023	ONE=1.Dr			
0024	DO 10 1=1			
3025		EO. ANT GO TO 16		
0056		E-AN+SN(I) I/LW		
0027	1) x=nS(1) -			
0028		ZRO1 GO TO 10		
(+02.9		OSTPITOX/LW: 102		
ዕ ዮ 3 በ	X=DSIN(PI	I+DX/LWI/DSORT(2.00-005	INE)	
0031	YI#ATAN(Y			
0032	/1 = ZODNE+	Y1		
0033	ZZA=ZONE#			
0534	Z 2 = Z 2 4 - Z 1		•	
0035	23=20NE-2	2A#71		
0036	/4=12/13			
0037	フッ=プのつNE♥	K)		
9038	76=74+75			
0039	77=200NE+	K2		
0040	2 6 ≥ 7 ONE + 7	.4 4 27		
6041	19= 16/1A			
0942	710=/30N8			
0043	711=210+7			
2044	711RE=SIL	LY(1)		
0045	7111H=SI			
0046		Z11RE**2+Z111M**2)		
0047		EO. 1) GO TO 141		
0048		GE. 21 GD TO 142		
0049		ONE.AND.ZllRE.GE.ZROL C		
<u> </u>		.OME.AND.ZIIRE.LT.ZRO) O		
0051		.ONE.AND.Z11RE.GE.ZRO) C		
0052		ONE.AND.Zlire.LT.ZRO) (
2053		ONE.AND.Z11RE.GE.ZPO) (
0054		.ONE.AND.JLIRE.LT.ZRO) (
0055		CNE.AND.711RE.GE.7RO1 C		
0056		.GNE.AND.Z11RE.LT.ZRO) (CO TO 173	
0057	150 R=ONE	•		
0058	A=42=U=1			
0659	CD 13 16)		
0060	151 6=22=0-1			
1900	A=2.0040	K/LW		

PROCRAM II (cont.)

```
0052
                   69 TO 160
6063
               157 BEGNE
0064
                   A=6.D3*DX/LW
0065
                   GO TO 160
0066
               153 R=18.D-1
0067
                   .A≃DX/LW
0068
                   GO TO 160
0069
               170 ND8=N(I)
0070
                   R=[ ND8-ONE] *PI[+7854; D-4
0071
                    4=4.07*DX/LW
0072
                   GO TO 160
0073
               171 NOS=NII)
0074
                   R=ND8#PII-7854.D-4
0075
                    A=4.DJ4DX/LW
0076
                    60 TO 160
0077
               172 ND8=N(1)
0078
                    B=f2.D04NUB-1.D01415768.D-4-1.D-1/ND8
0679
                    A=DX/LW
DORO
                    GO TO 160
DERI
               173 ND8=N(1)
2082
                    B=(2.03+ND8-1.D0)+15708.D-4+1.3-1/ND8
0083
                    A=DX/LW
0684
               160 TAHA=DTANH(A)
0,085
                    TANE = DTAN(B)
0086
                    A2=T AHA+10NF+TAN8++2)/(ONE+TAHA++29TAN8++2)
0087
                    82= TANR # ( DNE-T AHA##21/( DNE+TAHA##2#T ANR##2)
DURB
                    Z14RE={4*A2+B*B21/{A**2+B**2}
0089
                    7141M=1A+82-8+A21/(A++2+8++21
COSO
                    FRROR1=DSORT(|Z14RE-Z11RE)##2+(Z14IM-Z11IM)##2}
0991
                    DO 400 K=1.22
0092
                    SOLD=STEP(K)
0093
                    TAHA=DTANH(A)
               D9 600 J=1.2
420 WE=ONE+STEP(KI*F(J)
11194
C095
0696
                    KOUNT=0
               401 KOUNT = KOUNT+: ) 0098
0097
                                               1F (KOUNT.GT.10) GO TO 411
0049
                    GO fn 425
0130
               411 STEP(K)=STEP(K!*10.00
0103
                   60 TO 426
2102
               425 BOLD=B
0103
                    214100=71419
01/14
                    714R00=714RE
0105
                    FROILD=FRROR1
0106
                    B=B#WE
9107
                    TANS : DTAN(B)
6178
                    A7=TAHA+(ONE+TANB++2)/(ONE+TAHA++24TANB++2)
                    82=TAN9#(ONE-1A4A**2)/(ONE+TAHA**2*TAN8**2)
0119
                    714RE=(4442+8#82)/(4##2+8##2)
0110
0111
                    7141M={ 1#82-8#A21, (A0#2+6##21
0112
                    FPROR1 = DSORT (( 214RE- Z11RE) + +2+(214 IM-Z11 IM) + +2)
0113
                    (F(ERROR1-LE-ERGLD) GO TO 401
0114
                    714[M=21410D
                    7148E=Z14ROD
0115
                    丹金もりたり
0116
                    FRROR1=FROLD
9117
2118
                    STEPIKI=SOLD
0119
               600 CONTINUE
0120
                    TANB=DTAN(R)
0121
                    00 799 J=1.2
0122
               421 WE=ONE+STEP(K)*F(J)
0123
                    KCUNT=C
5124
               462 KOUNT=KOUNT+1
                    1F (KOUNT. GT. 19) GD TO 412
7125
0126
                    GO TO 428
               412 STEP(K)=STEP(K)+10.00
2127
012R
                    GO: TO 421
0129
               428 AOLD=A
0130
                    714RND=714RF
0131
                    714100=7141M
0132
                    EROLD=FRRORI
0133
                    A=A+WE
2134
                    TAHA=OTANH (A)
2135
                    AZ=TAHA#(ONE+TANB#+2)/(ONE+TAHA##2#TANB##2)
2136
                    RZ=TANP# (ONE-TAHA##21/(ONE+TAHA##2#TANR##2)
0137
                    714K5=[A#A2+B#B23/(A##2+B##2)
2138
                    7141M=14+B2-B#A21/1A##2+B##21
7139
                    FRRORL=DSORT((714RE-Z11RE)++2+(Z14IM-Z11IM)++2)
```

PROGRAM II (Cont.)

```
0140
                   IF(ERROR1-LEGEROLD) GO TO 432
3141
                   214RE= 214ROD
0142
                   7141M=714100
0143
                   A=AOLD
0144
                   ERROR1 = EROLD
2145
                   STEP(KI=SOLD
0146
               700 CONTINUE
0147
                   IF(ERROR1.LF.1.D-6) GO TO 450
0148
               400 CONTINUE
0149
               450 Z12RE=ZONE#A
                   Z12TM=ZNONE#B
0150
                   Z12NEW=(Z12RE+Z12IM)##2
0151
                   Z13=-Z12NEW+K3++2
2152
                   G=ZONE*FC
0153
0154
                   H=ZONE+W
0155
                   715=(G+2131/H
                   714R=70NE# 714RE
0156
                   Z14I=ZOONE#Z14IM
0157
0158
                   Z14=Z14R+Z14I
0159
                   RF=FAKE(1)
                   AIM=-FAKE(2)
0160
0161
                   TAM=ATM/RE
               3GD FORMAT(2X.F7.4.3X.F7.4.3X.12.3X.F7.4.3X.F9.6.3X.F9.6.3X.E13.6.
0162
                  22X.E13.6.3X.E13.6.2X.E13.6)
0163
                   WRITE(G.3CO) SN(I).DS(I).N(I).RE.AIM.TAM.Z11.Z14
0164
                10 CONTINUE
0165
                   GO TO 77
                88 CALL EXIT
0166
                   END
0167
```

TYPICAL PRINT-OUT

NS	os	N	K1	K2	TAN
7-0000	0.0100	1	1.1471	0.002666	0.002324
7-0202	1.0000	1	1.1138	0.453739	0.437376
6-5000	0.0100	1	1.7884	0.004175	0.002335
6- 5000	1.0000	1	1.5719	0.650037	0.413534
6-0000	0. G100	1	3, 2139	0.011904	0.003704
6.0000	1.0200	1	2.0000	1.525029	0.762,20
7-5000	0.0100	2	6.9674	0.001903	0.000273
7-5000	1.0000	2	6.9787	0.325143	0.046591
7-0202	0.0130	2	7.3665	0.302279	0.000309
7.0000	1.0000	2	7.2929	0.375678	0.051513
6.5000	0.0170	2	8.0271	C.005175	0.000645
6- 5000	1.0000	2	7.6436	D.681794	0.089198
6.0000	0.0100	2	11.0249	0.039429	O.CG3576
6.0000	1.0000	2	7.4015	1.728636	0. 233552

Z11 Z14

```
-0.2091410-01
0.241432D G1
               -0.209141D-U1
                                  0.2414320 01
0.8459020 00
               -0.9386930 00
                                  0.8459020 00
                                                 -0.938693D 00
-0.1543920 01
               -0.205383D-01
                                 -0.154392D 01
                                                 -0.2053830-01
0.3352290-02
                                  0.3352200-02
                                                 -0.935078D 00
               -0.9350780 00
                                 -0.1034650 00
0.7152920-01
0.103465D DG
                                                 -0.2770130-02
               -0.2770130-02
n. 715289D-01
               -0.393698D 00
                                                 -0.3936980 00
                                  0.957216D 00
0.786797D 00
               -0.2783620-02
                                                 -0.278363D-C2
0.9572150 00
                                                 -0.394979D 00
0.7807970 30
               -0.394979D 00
                                                 -0.2091410-01
0.2414320 01
               -0.2091410-01
                                  0.2414320 01
0.845902D 00
               -C. 938693D DO
                                  0.8459020 00
                                                 -0. 938694D CC
-0.154392D C1
                -0.2053830-01
                                 -0.154392D Ul
                                                 -0.2053830-01
0.3352290-02
                                  0.3351370-02
                                                 -0.935078D 00
               -G.935078D 00
                                                 -0.2770140-02
                                 -0.103466D 00
               -0.2770130-02
-0.133465D 00
0.7152890-01
               -C.393698D 00
                                  0.715288D-C1
                                                 -0.393698D 00
```

INDEX TO DIELECTRIC DATA

I. INORGANIC COMPOUNDS	Pag
Aluminum oxide, single crystal, Union Carbide	17
" multicrystalline, G.E. A-923	17
A-1004	17
Boron mitride, hot-pressed, Battelle Memorial Institute	18
Magnesium aluminate (spinel), single crystal, Union Carbide	18
Silica slipcast, Dynasil	18
Silica fiber, Philco Ford	18,19
Silica fiber phosphate, Whittaker Corp.	20
Silicon nitride, Admiralty Materials Lab.	20
II. MISCELLANEOUS INORGANICS AND MIXTURES	
Concrete pavement, California Highway Department	21
Asphalt pavement, " " "	21
III. ORGANIC COMPOUNDS	
(Listed according to source)	
Budd, copper-clad laminate	22,23
Carborundum, EKONOL (polyester resin)	23
Dodge Industries, FLUORGLAS E 650/2-1200	24
E.I. du Pont de Nemours, Nonex honeycombs	24
IV. LIQUIDS	
(Listed according to source)	
Hercules Inc., VUL-CUP, a,a ¹ -bis(t-butyl peroxy) diidopropybenzene	25
" DI-CUP, dicumyl peroxide	25
Pennwalt Corp., Lucidol Div., Lucidol, t-butyl perbenzoate	25
" Lupersol 130, 2,5-dimethyl-2,5-di(t-butylperoxy) hexyne-3	25
U.S. Peroxygen Div., Argus Chemical Corp., USP 333	26
Wallace & Tiernan Inc. Lupersol 101, 2.5-dimethyl-2.5-di(t-hutylneroxy)he	xane 26

I. INORGANIC COMPOUNDS

Aluminum oxide, single crystal

Union Carbide, Electronics Div.

*****--

<,0001

<.00015

Sapphire Al_2O_3 Density at $25^{\circ}C = 3.9840 \text{ g/cm}^3$

 $\boldsymbol{T^{O}}\boldsymbol{C}$

25

80

240

377

526

617

713

Freq. 3.45 - 3.33 GHz

E L C

κ tan δ

9.39 <.0001

9.41 <.0001

9.49 <.0001

9.62 <.0001

9.83 <.0001

9.95

10.08

Aluminum oxide, multicrystalline A-923 (97% ${\rm Al}_2{\rm O}_3$) Density 3.740 g/cm 3 General Electric Company A-1004 (94% Al₂0₃) Density 3.649 g/cm³

Freq. 3.74 - 3.37 GHz			Freq. 3.80 - 3.61 GHz		
κ	tan δ	т ^о с	κ	tan ô	
9.31	.00039	25	9.02	.00076	
9.41	.00042	100	9.11	.00078	
9.58	.00053	200	9.26	.00081	
9.72	.00070	300	9.40	.00093	
9.84	.00090	400	9.55	.00109	
9.96	.00112	500	9.69	.00128	
10.17	.00160	600	9.84	.00177	
10.42	.00215	650	9.92	.00335	
10.63	.00265	700	10.00	.0093	
10.86	.0033				
10.98	•0040				
11.17	.0045				
11.22	.0050				
13.38	.0060				
11,41	.010			•	
	9.31 9.41 9.58 9.72 9.84 9.96 10.17 10.42 10.63 10.86 10.98 11.17 11.22 138	k tan δ 9.31 .00039 9.41 .00042 9.58 .00053 9.72 .00070 9.84 .00090 9.96 .00112 10.17 .00160 10.42 .00215 10.63 .00265 10.86 .0033 10.98 .0040 11.17 .0045 11.22 .0050 1.38 .0060	κ tan δ T°C 9.31 .00039 25 9.41 .00042 100 9.58 .00053 200 9.72 .00070 300 9.84 .00090 400 9.96 .00112 500 10.17 .00160 600 10.42 .00215 650 10.63 .00265 700 10.86 .0033 10.98 .0040 11.17 .0045 11.22 .0050 1.38 .0060	κ tan δ T°C κ 9.31 .00039 25 9.02 9.41 .00042 100 9.11 9.58 .00053 200 9.26 9.72 .00070 300 9.40 9.84 .00090 400 9.55 9.96 .00112 500 9.69 10.17 .00160 600 9.84 10.42 .00215 650 9.92 10.63 .00265 700 10.00 10.86 .0033 .0040 .0045 11.17 .0045 .0050 138 .0060 .0060	

Boron nitride

Battelle Memorial Institute

(hot-pressed, after vacuum treatment)

Density in g/cm³

•	8.52 GHz,	25°C	4 €₹*
Sample	Density	κ	tan ô
115H7		4.37	.00030
118н7	2,132	4.87	.00025

Magnesium aluminate (spinel) ${\rm MgOAl}_2{\rm O}_3$

Union Carbide, Electronics Div.

Single crystal

Density at $25^{\circ}C = 3.57389 \text{ g/cm}^3$

Freq. 4.23 - 4.07 GHz

TOC	κ <u>÷</u> 02	tan ô
25	8.28	.0001
150	8.42	.0002
231	მ₊54	.0002
297	8.64	.0003
421	8.85	.0010
455	8.91	.0025

Silica, slip-cast

Dynasil Corp. of America

8.6 GHz, 25°C

Sample	Density (g/cm^3)	ĸ	tan δ
DSCX-3	1.970	3.395	.00058
DSCX-8E	2.038	3,513	.00054

Silica fiber composites

Philco-Ford Corp., Aeronutronic Div.

<u>Sample</u> 1-VH-O-M-1, 25 ⁰	'C	(Hz) 10 ⁵	10 ⁶	10 ⁷	7.5x10 ⁷	1.8x10 ⁸
As received,				2.777			2.772*
density 1.536 g/cm ³	104	tan δ	4.6	8.3	6.4	13.4	17*
After 18 hrs.		κ			2.77	2.77	2.77*
vacuum oven 80°C	104	$ \text{tan } \delta$			4.6	9.1	11.5*
*							

Extrapolated values.

Silica fiber composites (cont.)

Philco-Ford Corp., Aeronutronic Div.

Sample 1-XB-O-M			
Density 1.653 g/cm ³		8.52 GHz	
	т ^о с	κ	itan δ
As received, Face 1 up	25	2.919	.0062
Face 2 up	25	2.956	.0064
After vacuum oven			
80°C, 10 days			
Face 2 up	25	2.938	.00162
Face 1 up	25	2.895	.00169
	115	2.89	.0012
	246	2.89	.0006
	357	2.90	.0005
	438	1	.0006
	535		.0008
	608		.0010
	710		.0014
·	805	Ì	.0020
	908	.	.0026
	972	. }	.0028
	1000	♥ *	.0031*
	25	2.89	.00042

^{*} Extrapolated values.

			8.52 GHz		
	Sample	$\mathbf{r}^{\mathbf{o}}$ c	κ	tan ó	Density (c/cm ³)
	1, as received	25	2.73	.0051	1.547
	2, as "	25	2.79	.0060	1.543
	2, dried*	25	2.68	.0043	(wt. loss .049%)
	2, room humidity	25	2.70	.0050	
		116	2.70	.0050	
		235	2.71	.0053	
		410	2.71	.0080	
		495	2.71	.0105	
		580	2.72	.0140	
		673	2.72	.0177	
		760	2,72	.0228	
		327	2.73	.0265	
		916	2.74	.0315	
-		967	2.75	.038	
		25	2,71	.0047	

 $^{^{\}star}$ 4 days at $120^{\rm o}{\rm C}$ in vacuum oven.

Sil	licon	nitride	ceramic	
At	8.52	CHz . dei	nsiry 2.44	9 e/cm3

Admiralty Materials Laboratory

Hz, density 2.449 g/cm ³		
T ^O C	κ	tan δ
25	5.54	.0036
170	5.54	.00375
323	5.54	.0040
446	5.55	. 00365
586	5.55	.0030
674	5.56	•0050
714	5.57	•0054
864	5.58	.00615
912	5,59	.00630
991	5.63	.00665
509	5.55	.0034
348	5.54	.0040

11. SISCELLANEOUS INORGANICS AND MIXTURES

Concrete 1)aVefocut				California Hig	- hway Department
Sample S1 S1	Density Dry Wat	(Miz) κ tan δ κ tan δ	0.1 9.05 .0946 176.5 .822	1 7.97 .0913 69.2 1.088	10 .0736 23.5 .734	100 6.57 .0536 13.2 .485
Asphalt pa						
nophare pa	Vezent			•	California High	hway Department
Sample	Vement Denuity	(Rz)	10 ⁵	10 ⁶	California High 10 ⁷	hway Department
		(Hz)	10 ⁵ 4 . 51			
<u>Sample</u> S	Density Dry	κ tan δ	4.51 .0280	10 ⁶ 4.34 .0221	10 ⁷ 4.21 .0181	108
Sample	Density	κ tan δ κ	4.51 .0280 42.0	10 ⁶ 4.34 .0221 17.7	10 ⁷ 4.21 .0181 9.03	10 ⁸ 4.14
<u>Sample</u> S	Density Dry	κ tan δ	4.51 .0280	10 ⁶ 4.34 .0221	10 ⁷ 4.21 .0181	10 ⁸ 4.14 .0198

.0187

.368

14.48

.0158

9.28

.280

.0123

6.65

.190

.0121

6.01

.104

tan é

tan ò

κ

Wet

L

III. ORGANIC COMPOUNDS

	Copper-clad	laminate	PE115	3			The	Budd Co.,	Polychem	Division
	E <u>1</u>				102	10 ³	104	105	10 ⁶	10 ⁷
	3-terminal, liquid im- mersion, unclad*	25	κ tan	ô			2.420	ૺ		
	Declad †	25	κ tan	ô			2.451			
	3-terminal, clad	26	κ tan	δ	2.650 .0945	2.465 .0279	2.438 .00484	2.432 .00093		
		-195	κ tan	δ	2.416 .00030	2.415 .00033	2.414 .00036	2.411 .00022		
		-54	κ tan	δ	2.433 .00042	2.421 .00050	2.417 .00052	•		
	2-terminal, clad, meas. 12-21-70	25	K tan	δ	2,495 .01816	2.475 .00307	2.471 .00083	2.469 .00050	2.468 .00055	2.468 .00035
-	3-terminal, clad, 2nd	25	K tan	δ	2.843 .141	2.504 .0491	2.457 .00811	2.449 .00126		
	sample	96	K tan	δ	2.486 .0708	2.398 .01343	2.389 .00230	2.384 .00050		
		250	κ tan	δ	2.257 .0263	2.240 .00568	2.232 .00238	2.222 .00111		
		25	κ tan	δ	2.759 .0970	2.510 .0390	2.459 .00748	2.451 .00115		
	2-terminal, clad	-54	κ tan	δ	2.484 .00034	2.484 .00044	2.484 .00065	2.479	2.464 .00059	2.456 .00110
		-1 95	κ tan	δ	2.490 .00029	2.487 .00038	2.485 .00053	2.484 .00028	2.482 .00049	2.470 .00091
		25	κ tan	δ				2.462 .00050	-	2.458
		96	κ tan	δ	2.474 .01193	2.464 .00226	2.461 .00084	2.460 .00059	2.458 .00050	2.455 .00068
		250	κ tan	δ	2.333 .01013	2.319 .00340	2.312 .CO177	2.309 .00147	2.298 .00097	2.295 .00070
		25	κ tan	δ	2.422 .00995	2.415 .00189				
	E H									
	2-terminal, unclad	25	κ tan	δ	2.434 .0037	2.533 .00094	2.431 .00061	2.428 .00035	2.416 .00052	2.413 .0005

^{*} Refers to sheet stock received without copper.

Refers to a sample made by mechanically stripping the copper-clad sheet.

Resonant-Cavity Measurements:

 \sim 8.5 GHz, sample constrained in parallel direction, allowed to expand with temperature against a force 30 lb/sq in the perpendicular direction. Unclad stock.

	E.	L	E II		Thickness
$\mathbf{T}^{\mathbf{O}}C$	κ	tan δ	ĸ	tan δ	cm
-194	2.466	.00063	2.420	.00095	1.911
-54	2,437	.00070	2.397	.00104	1.917
23	2.421	.00091	2.383	.00130	1.924
96	2.396	.001:7	2.367	.00147	1.948
250	2.296	.0022	2.246	.001.85	2.093
Standi	Ing-wave metho	od, 25°C			
	Ell , one pie	ce unclad	2.387	.00128	

EKONOL (polyester resin)

The Carborundum Company

Frequency, 11z	r°c	к	tan ô
10 ²	25	3.21 i	.00289
10 ³		3.210	.00316
104		3,185	.00336
10 ⁵	ļ	3,168	.00348
10^6		3.156	.00325
107		3.148	.00220
108		3.140	.00215
8.5x10 ⁹	1	3.120	.00281
1	99	3,11	.0030
	155	3.08	.0040
	207	3.07	.0061
	284	3.04	.0104
	350	3.03	.0230
	420	3.03	.0230
	217	2,99	.0067
\	25	2.96	.0025

FLUORGLAS E 650/2-1200 TFE-fiberglas laminate

Dodge Indum des, Inc.

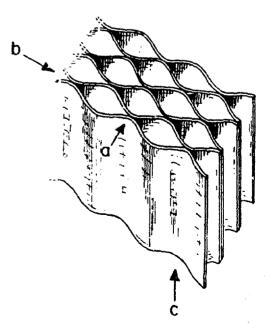
	Provide contract	n	I	: L	E	
	Freq., GHz	$\mathbf{r}^{\mathbf{o}}\mathbf{c}$	к	tan o	ĸ	tan o
	8.5	∴3	2.505	.0014	2.847	.0036
•	. 4	-195	2.533	.00082	2.896	.00172

Nonex honeycombs
At 8.52 GHz

E. I. du Pont de Nemours and Company

Sample No.	Density	a direction E L double-layer seam		b direction E n double-layer seam		c direction E holes	
		κ	tan 6	ĸ	tan δ	K	tan δ
1	1.398	1.0348	.00089	1.0441	.00141	1.0855	.00212
3.	2.892	1.0519	.00165	1.0669	.00229	1.0951	.00350
3	3.938	1.0788	.00176	1.1258	.00326	1,1444	.0041
4	4.039	1.0808	.00187	1.1020	.00278	1,1265	.0047
5	4.124	1,0827	.00274	1,1045	.00359	1.1351	.0046
6	4.259	1.0863	.00197	1.1340	.00382	1.1270	.0045
7	4.701	1.0928	.00315	1.1115	.00297	1.1455	.0047
8	5.603	1.0990	.00205	1.1781	.00468	1.1869	.0047
8*	5.603	1.1010	.00330	1.1667	.00628	1.1009	•0003

* At 100° C, all other v.lues at 25° C



IV. LIQUIDS

Hercules Inc.

•				nero	cules inc.
Ant-cab				PI-CUP	
a,a ¹ -bis(t-butyl	peroxy)	diisopropylbenzen	e	dicumv	l percxide
	25°C			25°C	
Freq., Hz	κ	tan ô		K	tan δ
10 ²	2.633	.0011		2.79	.0073
10 ³	1	.00011		1	.00081
10 ⁴		.000013		1	.000115
10 ⁵		10 ⁻⁵			.000064
10^6		.00005 <u>+</u> 2		Ţ	.00040
1.8x10 ⁷		_		2.97	.0032
6x1.0 ⁹	\rightarrow		•	2.73	.0025
10 ⁸	2.63	.005 <u>+</u> 2		2.70	.0050
109	2.60	.0206		2.57	.0082
3x10 ⁹	2.56	•0378		2.515	.0078
8.5x10 ⁹	2.40	.056		2,495	.0044
	99 ⁰ c				
10 ⁹	2.26	.0116			
3x10 ⁹	2.24	.0184			
			Per	nnwalt Corp., L	ucidol Div.
Lucidol		1	upersol 130		
t-butyl perbenzo	oate	2	2,5 wimethy1-2	,5-di(t-butylpe	roxy)hexyne-3
	25°C			25°C	
Freq., Hz	ĸ	tan δ	Freq., Hz	κ	tan ô
10 ²			10 ²	2.656	.00123
10 ³	-	==	10 ³		.000123
104	12.17	.17	104		.000012
10 ⁵	12.17	.017	105		.000023
106	12.1	.0027	106	\	.00012
107	12.0	.0095	107	2.655	.00121
108	11.2	.0044	108	2.65	.0066
10 ⁹	5.70	.252	109	2.56	.0235
3x10 ⁹	4.07	.337	3x10 ⁹	2.50	.0344
8.5x10 ⁹	3,23	.460	8.5x10 ⁹	2.39	•0505
				99 ⁰ C	
				2.33	•0076

2.32

.0154

U.S. Peroxygen Div., Argus chemical Corp.

Fraq., Hz	25 ⁶ C K	tan ô
102	3.818	0170
103		.00170
104		.00017
105		.000027
10 ⁶	\	.00021
10 ⁷	3.81	.00157
108	3.75	.0146
10 ⁹	3.60	.0842
3x10 ⁹	3.30	.130
8.5×10 ⁹	2.80	.1735

Lupersol 101
2,5-dimethy1-2,5-di(t-butylperoxy)hexane

Wallace & Tiernan Inc.

	25°C	
Freq., Hz	κ	tan δ
104	2.66	.000088
10 ⁵	2.66	.000144
10 ⁶	2.66	.000053
10 ⁷	2.65	.00049
108	2.64	.0050
10 ⁹	2,62	.0217
3×10 ⁹	2,58	.0387
8.5x10 ⁹	2.41	.057
2.4x10 ¹⁰	2.26	.045
	99°C	
109	2,02	.0040
3×10 ⁹	2.02	.0068